FORM PTO-1390 U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE ATTORNEY'S DOCKET NUMBER (REV. 10-95) TRANSMITTAL LETTER TO THE UNITED STATES. J6547(C) DESIGNATED/ELECTED OFFICE (DO/EO/US) U.S. APPLICATION NO. (If known, see 37 CFR § 1.5) CONCERNING A FILING UNDER 35 U.S.C. § 371 10/089648 PRIORITY DATE CLAIMED INTERNATIONAL APPLICATION NO. INTERNATIONAL FILING DATE PCT/FP00/09144 18 SEPTEMBER 2000 1 OCTOBER 1999 TITLE OF INVENTION ANTIPERSPIRANT COMPOSITIONS COMPRISING MICROEMULSIONS APPLICANT(S) FOR DO/EO/US MA. ZHUNING ET AL. Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information: This is a FIRST submission of items concerning a filing under 35 U.S.C. § 371. This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. § 371. 2 This express request to begin national examination procedures (35 U.S.C. §371(f)) at any time rather than delay 3. examination until the expiration of the applicable time limit set in 35 U.S.C. §371(b) and PCT Articles 22 and 39(l). A proper DEMAND for International Preliminary Examination was made by the 19th month from the earliest claimed 4. priority date. A copy of the International Application as filed (35 U.S.C. §371(c)(2)) is transmitted herewith (required only if not transmitted by the International Bureau). a. [] has been transmitted by the International Bureau. b. 🖂 is not required, as the application was filed in the United States Receiving Office (RO/US). A translation of the International Application into English (35 U.S.C. §371(c)(2)). Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. §371(c)(3)) 7 are transmitted herewith (required only if not transmitted by the International Bureau). have been transmitted by the International Bureau. h П have not been made, however, the time limit for making such amendments has NOT expired. c n have not been made and will not be made. A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. §371(c)(3)). An oath or declaration of the inventor(s) (35 U.S.C. §371(c)(4)). A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 10 \$371(c)(5)). Items 11. To 16. Below concern document(s) or information included: 11.

An Information Disclosure Statement under 37 C.F.R. §§ 1.97 and 1.98. An assignment document for recording. A separate cover sheet in compliance with 37 C.F.R. §§3.28 and 3.31 is 12. II included. A FIRST preliminary amendment. 13. A SECOND or SUBSEQUENT preliminary amendment. 14. (1) A substitute specification.

15.

A change of power of attorney and/or address letter.

16. ☐ Other items or information:

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The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 12-1155. Triplicate copies of this letter are enclosed.

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PATENT TRADEMARK OFFICE NOTE: Where an appropriate time limit under 37 C.F.R. § 1.494 or 1.495 has not been met, a petition to revive (37 C.F.R. § § 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

Respectfully submitted,

u Tellea

Attorney of Record Reg. #34,396

RM/mt (201) 840-2671

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PATENT #99-0080-HC Case #J6547(C)

Express Mail Label No.: ET 506 459 646 US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Ma et al.

Applicant: Deposited:

April 1, 2002

For:

Antiperspirant Compositions Comprising Microemulsions

Edgewater, New Jersey 07020 April 1, 2002

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents Washington, D.C. 20231

Sir:

With regard to the above-identified application filed concurrently herewith, please amend the following:

In the Claims:

Please enter the following amended claims:

- 3. (Amended) A composition in accordance with claim 1 characterised in that said antiperspirant salt is a zirconium salt complexed with aluminum salts having coordinated or bound water.
- (Amended) A composition in accordance with claim 1 characterised in that said 4. antiperspirant salt is present in the aqueous phase at from about 1 to about 60%.

- 6. (Amended) A composition in accordance with claim 1 characterised in that said aqueous phase further comprises a buffer, a glycol, a sugar, a cyclodextrin, a preservative, an antimicrobial, a chelating agent, a water-soluble polymer, an anticholinergic, a monovalent salt, a divalent salt, a trivalent salt, fragrances or mixtures thereof.
- (Amended) A composition in accordance with claim 1 cit said aqueous phase is present at about 1% to about 60%, more preferably at 5% to 30%, and most preferably at 10 to 25%.
- 8. (Amended) A composition in accordance with claim 1 characterised in that said cosmetic oil comprises esters, ethers, long chain alcohols, or ethoxylated alcohols, hydrocarbons, fatty acids, monoglycerides, diglycerides triglycerides, fragrances and volatile or non-volatile silicone fluids, and cholesterol.
- 10. (Amended) A composition in accordance with claim 8 characterised in that said non-volatile silicone is phenyl tris(trimethylsiloxy)silane.
- 13. (Amended) A composition in accordance with claim 1 characterised in that the cationic quaternary ammonium surfactant has the following structure:

$$/-(CH_2)_x$$
-CH₃

R-CO-NH- $(CH_2)_n$ -N⁺- $(CH_2)_z$ -CH₃
 A - $(CH_2)_z$ -CH₃

wherein n is one to six. x is zero to three y is zero to three z is zero to three with the proviso that $x+y+z \le 6$

A is any physiologically acceptable counter ion which does not adversely affect the composition, and more specifically A can be selected from the group consisting of chloride, bromide, ethosulfate, methyl sulfate, lactate, acetate, nitrate or sulfate.

Where R is a ricinoleic derivative:

CH3 (CH2) 5CH(OH) CH2-CH=CH- (CH2)7-

Or mixtures thereof.

15. (Amended) A composition in accordance with claim 1 characterised in that said cationic quaternary ammonium surfactant is present at 0.1% to 30%, more preferably at 1% to 30%, most preferably at 2% to 15%.

REMARKS

The present amendment is submitted to eliminate multiple dependencies and to correct minor typographical errors. The amendments were not intended to and should not be construed to have been made for any reasons related to patentability of the claims.

Attached hereto is a marked-up version of the changes made to the claims by the current amendment. The attachment is captioned "Version with Markings to Show Changes Made".

Respectfully submitted,

Rimma Mitelman

Registration No. 34,396 Attorney for Applicant(s)

RM/mt (201) 840-2671

VERSION WITH MARKINGS TO SHOW CHANGES MADE

- (Amended) A composition in accordance with claim 1 er-2-characterised in that said antiperspirant salt is a zirconium salt complexed with aluminum salts having coordinated or bound water.
- 4. (Amended) A composition in accordance with any—preceding—claimclaim 1 characterised in that said antiperspirant salt is present in the aqueous phase at from about 1 to about 60%.
- 6. (Amended) A composition in accordance with any preceding elaimclaim 1 characterised in that said aqueous phase further comprises a buffer, a glycol, a sugar, a cyclodextrin, a preservative, an antimicrobial, a chelating agent, a water-soluble polymer, an anticholinergic, a monovalent salt, a divalent salt, a trivalent salt, fragrances or mixtures thereof.
- (Amended) A composition in accordance with any preceding claimclaim 1 cit said aqueous phase is present at about 1% to about 60%, more preferably at 5% to 30%, and most preferably at 10 to 25%.
- 8. <u>(Amended)</u> A composition in accordance with any—preceding—claimclaim 1 characterised in that said cosmetic oil comprises esters, ethers, long chain alcohols, or ethoxylated alcohols, hydrocarbons, fatty acids, monoglycerides, diglycerides triglycerides, fragrances and volatile or non-volatile silicone fluids, and cholesterol.
- 10. (Amended) A composition in accordance with claim 8 er-9-characterised in that said non-volatile silicone is phenyl tris(trimethylsiloxy)silane.

13. (Amended) A composition in accordance with any-preceding-claimclaim 1 characterised in that the cationic quaternary ammonium surfactant has the following structure:

wherein n is one to six.

x is zero to three

y is zero to three

z is zero to three

with the proviso that $x+y+z \le 6$

A is any physiologically acceptable counter ion which does not adversely affect the composition, and more specifically A can be selected from the group consisting of chloride, bromide, ethosulfate, methyl sulfate, lactate, acetate, nitrate or sulfate.

Where R is a ricinoleic derivative:

CH₃ (CH₂) ₅CH(OH) CH₂-CH=CH- (CH₂)₇₋

Or mixtures thereof.

15. (Amended) A composition in accordance with any preceding claimclaim 1 characterised in that said cationic quaternary ammonium surfactant is present at 0.1% to 30%, more preferably at 1% to 30%, most preferably at 2% to 15%.

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ANTIPERSPIRANT COMPOSITIONS COMPRISING MICROEMULSIONS

5 Field of the Invention

This invention is related to microemulsions that contain cosmetically active ingredients. In a preferred embodiment, this invention is related to antiperspirant salt-containing microemulsions that are stable, clear liquids and are easy and inexpensive to produce.

Background of the Invention

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The microemulsions of the present invention contain water. Microemulsions of the present invention are transparent or translucent, optically isotropic and thermodynamically stable mixtures of oil and water 20 stabilized by surfactants and perhaps co-surfactants. The particle size of the dispersed phase of a microemulsion is about 100 to about 2000 angstroms, more preferably are about 100 to about 1000 angstroms. They can form spontaneously or with a little energy. Therefore they are simple to prepare 25 and are not process dependent i.e. the order of addition of starting materials or speed / type of mixing is not critical to the preparation of the microemulsions. It would be desirable to formulate antiperspirant compositions using microemulsions because microemulsions are easy and inexpensive to process and can be inherently clear without

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requiring refractive index matching of the aqueous and nonaqueous phases.

Microemulsions have attracted considerable technological and scientific interest. Water-in-oil (w/o) microemulsions containing water, an ionic surfactant, a cosurfactant and oil are the most investigated. The ionic surfactant- containing microemulsions usually exhibit stability over a large range of temperature. When inorganic salts are added, the minimum surfactant level to form water-in-oil microemulsions will increase. As the hydrocarbon oil chain length increases, the solubilization of aqueous phase into the oil phase decreases, while the liquid crystal area increases. Nonionic surfactant-containing water-in-oil microemulsions require a large amount of surfactant as well. Unfortunately, nonionic surfactant-containing microemulsions commonly exhibit a small temperature range of stability

Microemulsions exist in the following forms: as waterin-oil, oil-in-water or as a bicontinuous phase, which is
also called the surfactant phase. As used herein, the term
"microemulsion means water-in-oil, oil-in-water or a
bicontinuous phase, or mixtures thereof. Bicontinuous phase
microemulsions are found to solubilize a high amount of
water and oil with lower levels of surfactant. The region
around a bicontinuous phase microemulsion may transition
into a swollen lamellar phase, otherwise known as a liquid
crystal phase, and in certain cases these phases
(microemulsion and liquid crystal) may co-exist. These
phases exhibit birefringence, shear induced (streaming)

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birefringence, and are thixotropic, viscoelastic and transparent. Because some of these systems exhibit increased viscosity the technical literature may refer to them as microemulsion gels.

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It is an object of the present invention to provide antiperspirant compositions, which contain high levels of antiperspirant salts, cosmetic oils and surfactants suitable for application to the axilla. It is also an object of the present invention to provide antiperspirant compositions that do not require refractive index matching of the aqueous and nonaqueous phases in order to be clear. It is also an object of the present invention to provide microemulsion antiperspirant compositions that require little energy to manufacture. These and other objects of the present invention will become more readily apparent in the present application.

Patents and patent documents, which are cited in connection with the disclosed invention, are as follows:

DE 196 42 090 A1 discloses cosmetic or dermatologic compositions based on microemulsions.

- U.S. Patent 5,162,378 discloses water in oil microemulsions comprising cetyl dimethicone copolyol, water, silicone, alcohol, and 5-40% by weight of one or more salts.
- U.S. Patent 5,705,562 discloses a method of
 spontaneously forming a highly stable clear microemulsion by
 combining water, a volatile cyclic methyl siloxane or a

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volatile linear methyl siloxane and a silicone polyether surfactant. U.S Patent 5,707,613 is in the same patent family as the just mentioned patent.

5 W0 94/22420 is concerned with silicone-based skin care products, which are applied to the skin as aerosols and form a clear gel on the skin.

WO 94/19000 discloses pharmaceutical compositions in the form of a microemulsion which comprise and oil, a mixture of high and low HLB surfactants in which the high HLB surfactant comprises an aliphatic, aryl or aliphaticaryl sulfate or sulfosuccinate or salt thereof, an aqueous phase and a biologically active agent.

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WO 94/08610 discloses pharmaceutical compositions in the form of microemulsions which comprise an oil, a mixture of high and low HLB surfactants in which the high HLB surfactant comprises a medium-chain fatty acid salt, an aqueous phase and a biologically active agent.

U.S. 5,575,990 discloses roll-on antiperspirant compositions which are clear and, when applied to the human skin, do not leave a visible white residue after drying. The clear antiperspirant roll-on compositions are stable under varying temperature conditions and provide a suitable cosmetically acceptable feel or sensation when applied to the human skin

30 U.S. 5,487,887 discloses roll-on antiperspirant compositions and more particularly concerns antiperspirant WO 01/24766 PCT/EP00/09144

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compositions which are clear and stable under varying temperature conditions and, when applied to the human skin, do not leave a visible white residue after drying. The compositions in the form of an oil-in-water microemulsion, comprise an antiperspirant active 5-30, PEG-7-glyceryl cocoate 5-25, emollients 0.5-3, cyclomethicone 3-7, and water 53-60%

10 Summary of the Invention

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The invention relates to a composition in the form of a microemulsion comprising an antiperspirant salt, a cosmetic oil, and a combination of at least one cationic quaternary surfactant and at least one nonionic surfactant.

Detailed Description of the Invention

The present invention is directed to antiperspirant salt-containing microemulsions that are stable and clear liquids, or clear antiperspirant gels.

Stable clear microemulsions containing cosmetic oils,

25 antiperspirant salt, water, quaternary surfactants and
nonionic surfactants have been discovered. The
microemulsions are primarily composed of bicontinous phase
but the compositions include water-in-oil, oil-in-water, and
microemulsion gels (liquid crystals). The microemulsions

30 are novel antiperspirant compositions that can be used in
different types of applicators such as roll-on, sponge,

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mousse, pad, wipe, brush, gel and aerosol or non-aerosol spray applicators.

The microemulsions discovered in this invention contain inorganic salts such as antiperspirant salts and cosmetic oils and the solubilization of high levels of both oil and aqueous solution of salts is achieved by incorporating combinations of a quaternary ammonium surfactant and a nonionic surfactant.

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More specifically, the invention relates to a composition in the form of a microemulsion comprising an antiperspirant salt, cosmetic oils, and a combination of at least one cationic quaternary surfactant and at least one nonionic surfactant.

The invention also relates to a method for controlling or preventing underarm perspiration and malodor, which comprises applying to the underarm area a composition according to the invention.

The characteristics of the microemulsions of this invention include one or more of:

- 25 The microemulsions exhibit stability over a relatively large range of temperature.
 - The viscosity ranges from a thick gel to a low viscosity sprayable liquid.
- The types of the microemulsions formed are dependent on the ratio of aqueous phase to the nonionic surfactant(s) and oil. When the percentage of the salt solution

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containing quaternary surfactant increases, the microemulsion changes from water-in-oil to oil-in-water type, and a bicontinuous microemulsion phase, or possibly a liquid crystal phase, will form in-between.

- The microemulsions can contain a high level of inorganic salts.
 - The microemulsions contain a quaternary surfactant and a nonionic surfactant.
 - · The microemulsions contain cosmetically acceptable oils.
- 10 A method for controlling or preventing underarm perspiration and malodor, which can be applied to the underarm area.
 - The application of the microemulsions can be accomplished by using various product dispensers.

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As used herein % means weight percent unless otherwise specified.

As used herein the term cationic surfactant means 20 guaternary ammonium surfactant.

The starting materials set forth herein are either known or can be prepared according to known methods. The compositions of the invention can be made by known methods or by methods that are analogous to known methods.

As used herein, microemulsions mean stable clear microemulsions containing cosmetic oil; antiperspirant salts, water and surfactants. The microemulsions described herein are primarily composed of bicontinous phase but the

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compositions can include water-in-oil microemulsions. The compositions of the invention can also comprise a liquid crystal (that is, a microemulsion gel). More specifically, the compositions of the invention are selected from the group consisting of a microemulsion, a liquid crystal (that is, microemulsion gel), or a mixture of a microemulsion and a liquid crystal. The compositions of the invention comprise an antiperspirant salt, a cosmetic oil, and a combination of at least one cationic quaternary surfactant and at least one nonionic surfactant.

The compositions of the invention are novel antiperspirant compositions that can be used in different types of applicators such as roll-on, sponge, mousse, pad, brush, wipe, gel and aerosol or non-aerosol spray applicators.

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All of the microemulsion compositions described contain antiperspirant salts and are clear and stable over a larger temperature range from room temperature to 45°C-50°C. The viscosity of some of the water-in-oil microemulsions are less than 10cst, therefore they are spray-able.

The invention relates to a composition in the form of a microemulsion comprising an antiperspirant salt, cosmetic oils, and a combination of at least one cationic quaternary surfactant and at least one nonionic surfactant.

A description of the ingredients included in the 30 compositions of the invention now follows. - 9 -

Antiperspirant Salts

Antiperspirant salts contained in these microemulsions include, but are not limited to, aluminum chlorohydrate, aluminum dichlorohydrate, aluminum sesquichlorohydrate, aluminum chlorohydrex propylene glycol complex, aluminum dichlorohydrex propylene glycol complex, aluminum sesquichlorohydrex propylene glycol complex, aluminum chlorohydrex polyethylene glycol complex, aluminum dichlorohydrex polyethylene glycol complex, aluminum sesquichlorohydrex polyethylene glycol complex, aluminum zirconium trichlorohydrate, aluminum zirconium tetrachlorohydrate, aluminum zirconium pentachlorohydrate, aluminum zirconium octachlorohydrate, aluminum zirconium trichlorohydrex glycine complex, aluminum zirconium tetrachlorohydrex glycine complex, aluminum zirconium pentachlorohydrex glycine complex. aluminum zirconium octachlorohydrex glycine complex, aluminum chloride or buffered aluminum sulfate.

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Antiperspirant actives for use herein are often selected from astringent active salts, including in particular aluminum, zirconium and mixed aluminum/zirconium salts, including both inorganic salts, salts with organic anions and complexes. Preferred astringent salts include aluminum, zirconium and aluminum/zirconium halides and halohydrate salts, such as chlorohydrates.

Aluminum halohydrates are usually defined by the general formula Al $_2$ (OH) $_{\rm x}Q_{\rm y}$ or a hydrate thereof in which Q represents chlorine, bromine or iodine, x is variable from 2

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to 5 and x+y=6 . The level of hydration is variable for example wherein there are up to about 6 or higher water molecules.

5 Zirconium actives can usually be represented by the empirical general formula: ZrO (OH) 2n-nzBz or a hydrate thereof in which z is a variable in the range of from 0.9 to 2.0 so that the value 2n-nz is zero or positive, n is the valence of B, and B is selected from the group consisting of chloride, other halide, sulphamate, sulfate and mixtures 10 thereof. Possible hydration to a variable extent is represented by wH2O. It is preferable that B represents chloride and the variable z lies in the range from 1.5 to 1.87. In practice, such zirconium salts are usually not employed by themselves, but as a component of a combined 15 aluminum and zirconium-based antiperspirant. The level of hydration is variable for example wherein there are up to about 6 or higher water molecules.

The above aluminum and zirconium salts may have coordinated and/or bound water in various quantities and/or may be present as polymeric species, mixtures or complexes. In particular, zirconium hydroxy salts often represent a range of salts having various amounts of the hydroxy group.

Zirconium aluminum chlorohydrate may be particularly preferred.

Antiperspirant complexes based on the above-mentioned astringent aluminum and/or zirconium salts can be employed. The complex often employs a compound with a carboxylate group, and advantageously this is an amino acid. Examples

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of suitable amino acids include d1-tryptophan, d1-βphenylalanine, dl-valine, dl-methionine and β -alanine, and preferably glycine, which has the formula CH2 (NH2) COOH.

Complexes of a combination of aluminum halohydrates and zirconium chlorohydrates with or without with amino acids such as glycine can be employed in this invention. Certain of those Al/Zr-glycine complexes are commonly called ZAG in the literature. Aluminum-Zirconium actives or ZAG actives 10 generally contain aluminum, zirconium and chloride with an Al/Zr ratio in a range from 2 to 10, especially 2 to 6, an Al/Cl ratio from 2.1 to 0.9. ZAG actives also contain a variable amount of glycine. In certain conditions, salts with an Al/Zr ratio greater than 2 (also known as lowzirconium actives) may be preferred. Actives of these preferred types are available from Westwood, from Summit and from Reheis.

Other antiperspirant-salt actives that may be utilized include astringent titanium salts, for example those 20 describe in GB 2299506A.

The proportion of solid antiperspirant salt in a composition normally includes the weight of any water of hydration and any complexing agent that may also be present in the solid active. However, when the salt is in solution, its weight excludes any water present.

The antiperspirant active will often provide from 1 to 60% by weight of the aqueous phase, particularly from 10% to 30 60% of the aqueous phase. The final content of the salts in

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the formulations can range from 0.1% to 40% but 5-35% is preferred.

Other Aqueous Phase Ingredients

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In addition to aluminum salts, the microemulsions, discovered in this invention, could solubilize aqueous solutions of monovalent, divalent and trivalent salts. The salts include sodium chloride, sodium sulfate, calcium chloride, calcium sulfate, magnesium chloride, aluminum sodium lactate, and mixtures thereof.

Other ingredients which can be dissolved in the aqueous phase include buffers, glycols, sugars, cyclodextrins, preservatives, antimicrobials, fragrances, chelating agents, amino acids, antimicrobials, anticholinergics, water-soluble polymers etc.

Water Content

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The antiperspirant salts or other aqueous phase ingredients can be dissolved into water first and then combined with the non-aqueous phase. Water content in the final formulations can range from 1% to 60%, 5% to 30% is preferred and 10% to 25% is the most preferred.

Oil Phase

The oil phase of the compositions of the invention can contain cosmetic oils such as esters, ethers, long chain alcohols or ethoxylated alcohols, hydrocarbons, fatty acids,

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monoglycerides, diglycerides or triglycerides, fragrances, volatile or non-volatile silicone fluids. Cholesterol and some other lipids can be incorporated with the oil phase to act as emollients. The oil phase concentration can range from 0% to 95%, but 20% to 60% is preferred.

Silicone fluids that may be included in compositions of the invention include volatile and non-volatile silicone fluids such as cyclomethicones and dimethicones.

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Non-volatile silicones such as phenyl tris(trimethylsiloxy) silane can be included in compositions of the invention.

Silicone elastomers such as DC 9040, or DC 9010 by Dow Corning or GE SFE 839 by General Electric, can be included in the compositions of the invention.

Esters selected from the group consisting of cetyl

cotanoate, C12 -15 alcohol benzoate, isostearyl benzoate,
diisopropyl adipate, isopropyl palmitate, isopropyl
myristate and mixtures thereof may be included in the
compositions of the invention.

25 Hydrocarbon oils such as aliphatic hydrocarbons
(Permethyl 102A ™, Permethyl 101™); hydrogenated
polybutenes; hydrogenated polydecenes (Silkflo™);
dioctylcyclohexane; mineral oil, cyclohexane and mixtures
thereof may be included in the compositions of the
30 invention.

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Surfactants

Quaternary Ammonium Surfactants

5 Combinations of a cationic, quaternary ammonium surfactant(s) and a nonionic surfactant are employed in the compositions of the invention.

The quaternary surfactant in this invention is

10 essential, without which the formulation will be either
extremely sensitive to temperature or a microemulation will
not form. The preferred cationic surfactants employed in
compositions of the invention are alkylamidopropyl
alkyldimonium quaternaries.

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The preferred cationic quaternary surfactants have the following structure:-

/- (CH₂)
$$_x$$
-CH₃
20 R-CO-NH- (CH₂) $_n$ -N⁺- (CH₂) $_z$ -CH₃ A⁻

$$^{-}$$
 (CH₂) $_z$ -CH₃

25 y is zero to three

z is zero to three

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with the proviso that x+y+z ≤ 6

A is any physiologically acceptable counter ion which does not adversely affect the composition, and more specifically A can be selected from the group consisting of chloride, bromide, ethosulfate, methyl sulfate, lactate, acetate, nitrate or sulfate.

where R is a ricinoleic derivative:

10 CH₃ (CH₂) ₅CH(OH) CH₂-CH=CH- (CH₂) ₇-; or mixtures thereof.

Obviously, variations on this structure, known to the art, can also be incorporated into embodiments of this invention. The variations on surfactant structure should exhibit solubility in the aqueous antiperspirant salt solution. If the above mentioned solubility is maintained then variations in the quaternary ammonium salts can include but are not limited to, increasing or decreasing the alkyl chain length, changing the position or removal of the hydroxyl group, changing the position or removing completely the double bond or combinations thereof.

The most preferred quaternary surfactant is

25 ricinoleamidopropyl ethyldimonium ethosulfate a compound according to the formula above wherein n=3, x=1, y=0, z=0, A⁻

= ethosulfate and

 $R = CH_3 (CH_2) _5CH (OH) CH_2 - CH = CH - (CH_2) _7 - .$

30 The surfactant described just above is also known, under the following trade names, as Surfactol Q4 from WO 01/24766 PCT/EP00/09144

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CasChem Inc., Lipoquat R from Lipo Chemicals or Mackernium DC-159 from McIntyre Chemical. Preferably the quaternary surfactant is supplied in a concentrated form (>90% active) with a low free amine content. This form is readily miscible with the aqueous antiperspirant-salt solution.

The quaternary surfactant(s) in the compositions of the invention range from 0.1% to 30%, where 2% to 15% is preferred.

Nonionic Surfactants

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The nonionic surfactant or co-surfactants employed in the compositions of the invention can be polyethoxylated alcohol ethers or esters, polyglycerol mono or di-esters, glyceryl esters or branched guerbet ethoxylates or alcohols, or long chain carboxylic acids or combinations thereof. These compounds have a hydrophilic-lipophilic balance of between about 2 to about 15 and preferably less than about 12. Non-limiting examples are polyglycerol-3 diisostearate; glycerol oleate; poly glycerol-2 monoisostearate; polyglycerol -2 diisostearate, glyceryl isostearate. The most preferred ones are polyglyceryl-3 diisosterate, glyceryl isosterate and glycerol oleate or combinations thereof.

The ratio of cationic surfactant to aqueous phase containing antiperspirant salt ranges from 30/70 to 4/96, the ratio from 10/90 to 5/95 is preferred. The ratio of aqueous phase including salts, water and cationic surfactant

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to nonionic surfactant is 90/10 to 70/30, and the ratio from 90/10 to 80/20 is preferred.

Formulation Examples

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The following samples are stable for one month at room temperature. The particle size or domain length of these compositions are between about 150 to about 600 angstroms. All samples are clear. Some samples exhibit streaming birefringence. Some samples exhibit birefringence. The viscosity of these samples range from a thin liquid to a gel. These microemulsions are primarily composed of bicontinous phase but the compositions include water-in-oil, and microemulsion gels (liquid crystals).

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The following formulation examples are illustrative of the invention.

The following is a general formula for an 20 antiperspirant microemulsion of the present invention.

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General Formulation Example:

Componen	ts	Specific Examples	Range	Preferre
		of components		d range
Oil Phas	e*	Aliphatic		
		Hydrocarbon 90-10%	0-95%	20-60%
1		Volatile Silicone		
		10-90%		
Aqueous	Water	Deionized Water	1-60%	5-30%
Phase*				
	Antipers	ACH or AZG or	0.1-	5-35%
	pirant-	other salts	40%	
	Salt			
Non-ioni	С	Polyglycerol-3	0.2	4-15%
surfacta	nt	diisosterate	to	5-10% most
			30%	preferred
Cationic		Ricinoleamidopropyl	0.1-	2-15%
Quaternary		ethyl dimonium	30%	
Ammonium		ethosulfate		
Surfacta	nt			

*Cosmetic additives or other optional ingredients can be added to either phase as required.

Generalized manufacturing procedure:

10 1. Weigh all the oil phase components into a suitable vessel and mix until homogenous. Heat may be used to expedite dispersion of components solid at room temperature. - 19 -

- The aqueous phase is prepared by mixing the quaternary ammonium surfactant with the antiperspirant salt solution.
- Add the oil and water phases together and mix until a clear, homogenous dispersion is formed.
- 5 4. The microemulsion formulation is transferred into a suitable dispenser or applicator.

The following examples more fully illustrate embodiments of this invention, all percentages being by weight unless otherwise noted. The following specific examples, which are compositions of the invention, were made.

Compositions were prepared according to the following procedure:

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- Mix the cationic surfactant with the antiperspirant salt solution
- Mix the nonionic surfactant with the oil mixture, then add the two mixtures together and mix well.
- 3. Heat may be applied to better dissolve solid nonionic surfactants, which are solid such as glyceryl oleate, in the oil phase prior to mixing the aqueous and nonaqueous phases

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4.

Prisorine 3700 Cationic Alumin Water% DC245% HC* um Zircon ium tetra	
1 10 02 5 09 13 55 20 31 15 04 35 09	
10.03 3.96 13.35 20.31 13.04 33.05	
2 8.99 4.66 10.57 15.85 17.98 41.95	
3 7.02 3.45 7.82 11.74 20.99 48.98	
4 3.97 1.73 3.93 5.91 25.34 59.12	
5 Prisorine 3700 Cationic ACH Water DC245% HC	
8 * 8 8	
% % 6 9.97 6.78 19.2 19.2 13.45 31.40	
7 2.99 1.02 2.89 2.90 27.06 63.14	
1.02 2.03 2.700 33.14	-+
Glyceryl oleate Cationic Alumin Water DC 245 HC	
% Zircon ium terra	
8 14.24 11.71 22.09 33.13 5.65 13.18	
9 11.05 8.55 16.13 24.20 12.02 28.05	
10 10.02 7.89 14.88 22.33 13.46 31.42	
	irefri
	ngent
12 14.95 12.27 23.13 34.69 4.49 10.47	
Glyceryl oleate Cationic ACH Water DC 245 HC	
* * * * * * *	
13 3.99 12.91 36.57 36.57 2.99 6.97	
	irefri
	ngent
15 8.50 7.70 21.82 21.82 12.05 28.11	
Prisorine 3700 Cationic Alumin Water DC245% HC	
% ** um % * Zircon %	1
1 0 211001	İ
ium	1
penta	
penta 8 6 34.8 5.01 11.68 B3	irefri
penta	ngent
Penta	

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_							
	Glyceryl	Cationic **		Water %	DC 245	HC	
	isostearate	**	um	*	*	*	1
1	*	. *	Zircon ium			•	
			penta				
			benca penca				ŀ
19	11.02	11.09	25.15	37.72	4.51	10.51	Birefri
1	11.02	11.05	22.12	5,1.2			ngent
20	10.02	8.99	20.37	30.55	9.02	21.05	Birefri
1						ļ.	ngent
21	9.03	7.64	17.32	25.99	12.00	28.02	Birefri
							ngent
22	7.97	6.32	14.32	21.47	14.98	34.94	
23	6.02	3.60	8.15	12.22	21.00	49.01	
	Glyceryl	Cationic	Alumin	Water	DC 245	HC	
	isostearate	**	um	ક	8	*	
	*	*	Zircon			*	
		l	ium			1	l
			penta				
			*				
24	6.02	4.434	7.82	11.72	21.00	49.01	
25	8.52	13.64	24.03	36.05	5.33	12.43	
26	9.00	5.71	8.72	16.46	18.03	42.08	
26	4.68	0.14	0.25	0.38	28.36	66.19	
27	9.74	0.46	0.81	1.21	26.33	61.45	
\vdash	Glyceryl	Cationic	Alumin	Water	DC 245	HC	
1 1	isostearate	**	um	&	8	*]
1 1	용	*	Zircon			*	
1			ium				
1 1			penta				
L.			8		2.95	6.89	Birefri
28	11.47	11.80	26.76	40.13	2.95	6.89	ngent
-	11.11	11.07	25.10	37.65	4.52	10.55	ngenc
29				22.93	13.50	31.51	
30	10.03	6.74	15.29				
31	9.54	6.06	13.73	20.60	15.02	35.05	
32	11.38	11.91	27.00	40.51	2.76	6.44	
	Glyceryl	Cationic	Alumin	Water	DC 245	Silkofl	
	isostearate	**	um	8	*	0 366-NF	
	*	*	Zircon			366-NF	1
			ium		l	*	
]	penta %			l	l
33	7.45	16.94	30.34	44.66	0.43	0.18	Birefri
1		1 -5.54	30.54	11700		1	ngent
				33.59	13.85	5.92	Birefri
34	12.36	11.88	22.40	33.59	13.05	3.32	
34	12.36	11.88	22.40	33.59	13.05	3.32	ngent
34	12.36	11.88	22.40	33.59	13.89	5.95	
35	12.06	11.92	22.47	33.71	13.89	5.95	

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	Prisorine 3700	Cationic	Alumin	Water	DC 245	Silkflo	
	*	**	um	*	*	366-NF	
1 1		8	Zircon			*	
			ium				
1			penta				l l
			*				
38	10.67	11.19	25.36	38.05	10.31	4.42	Birefri
\vdash	1.31						ngent
39	14.01	9.89	22.41	33.61	14.06	6.02	
40	4.93	2.22	5.03	7.55	56.20	24.07	
41	13.98	6.90	15.64	23.45	28.02	12.01	
42	11.51	5.77	13.08	19.62	35.02	15.00	
43	9.51	4.58	10.37	15.56	41.98	18.00	
44	. 7.98	3.32	7.52	11.28	48.93	20.97	
	Prisorine 3700	Cationic	Alumin	Water	DC 245	Silkflo	
	*	**	um	8	*	366-NF	1
		*	Zircon			*	
			ium				
			penta				
			*				
45	11.05	13.48	25.42	38.08	8.34	3.63	
46	12.03	11.91	22.46	33.70	13.92	5.98	
47	11.96	9.80	18.49	27.73	22.41	9.61	Birefri
							ngent
48	15.96	11.22	21.16	31.73	13.95	5.98	Birefri
							ngent
49	14.03	9.78	18.44	27.66	21.06	9.03	
	Isofol 12	Cationic	ACH %	Water	DC245%	HC	
	alcohol	**		8		*	
	ethoxylate/	ક				*	
	cholesterol						
50	20.15/0	8.21	23.26	23.26	7.52	17.60	Birefri
							ngent
51	12.71/2.44	6.72	18.85	18.85	12.12	28.31	

- $\star~$ HC means hydrocarbon: Permethyl 102A, listed in the above table
- ** Cationic means the cationic surfactant:
- 5 Ricinoleamidopropyl ethyldimonium ethosulphate Further examples include: Example 52

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Ingredient (INCI)		Trade Name	28	Source	Percent
Ricinoleamidopropyl		Surfactol	Q4	CasChem,	7.50%
Dimonium Ethosulfate				Inc	
Polyglycerol-3		Prisorine	PG3	Uniqema	10%
Diisostearate		DI 3700			
Aliphatic Hydrocarbon		Permethyl	102A	Presperse	28%
Cyclopentasiloxane		DC245		Dow	12%
-				Corning	
Aluminum Chlorohydrate	50%	Westchlor	200	Westwood	42.50%
				Total:	100%

Example 53

Ingredient (INCI)	Trade Names	Source	Percent
Ricinoleamidopropyl	Surfactol Q4	CasChem,	7.50%
Dimonium Ethosulfate		Inc	
Glyceryl Isostearate	Peceol	Gattefoss	10%
	Isostearique	e	
Hydrogenated Polydecene	Silkflo 366	Lipo	12%
-		Chemicals	
Cyclopentasiloxane	DC245	Dow	28%
-		Corning	
Aluminum Zirconium	Low Zirconium	Reheis	42.50%
Pentachlorohydrate 40%	Penta]	
-	Solution R280-		
	130		
		Total:	100%

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Example 54

Ingredient (INCI)	Trade Names	Source	Percent
Ricinoleamidopropyl	Surfactol Q4	CasChem,	2.77%
Dimonium Ethosulfate		Inc	
Aluminum Zirconium	Low Zirconium	Reheis	47.63%
Pentachlorohydrate 40%	Penta Solution		
-	R280-130		
Glyceryl Isostearate	Peceol	Gattefoss	3.06%
-	Isostearique	e	
Hydrogenated Polydecene	Silkflo 366	Lipo	11.70%
1		Chemicals	
Cyclopentasiloxane	DC245	Dow	27.04%
		Corning	
Ethoxylated Guerbet	Novel II Isofol	Condea	7.80%
Alcohol C14 / 4 EO HLB ~9	14T+4E0	Vista	
		Total:	100%

5 Examples 55 and 56

Ingredient (INCI)	Trade Names	Supplier	55	56
			Per-	Per-
			cent	cent
Ricinoleamidopropyl	Surfactol Q4	Caschem	2.32	2.83
ethyl dimonium				
ethosulfate				
Aluminum zirconium	Rezal 67	Reheis	15.94	18.13
penta cholorohydrate				
Water	Deionized Water	Stock	23.91	27.19
Urea	Urea	Janssen	-	3.34
		Chimica		
Cyclopentasiloxane	DC 245	Dow	29.08	22.08
		Corning		
Polydecene	Silkflo366NF	Lipo	11.62	9.46
hydrogenated		Chemicals		
Glyceryl isostearate	Peceol	Gattefosse	5.26	-
	isostearique			
Polyglyceryl-3	Prisorine 3700	Unichema	0.87	3.49
diisostearate				
Ethoxylated Guerbet	Novel II I18T-	Condea	3.36	6.19
alcohol C18EO10	10 ethoxylate	Vista		
2-hexyldecanol	Isoflo 16	Condea	7.64	7.29
(Guerbet C16		Vista		
Alcohol)				
		Total	100	100

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Raw materials used in preparation of the example compositions of the invention are as follows:

Trade Name	Chemical Name	Vender
DC 245	Cyclomethicone D5	Dow Corning
DC 344	Cyclomethicone D4	Dow Corning
Silkflo 364 or 366	Hydrogenated	Lipo Chemical
	Polydecene	
Permethyl 102 A	Aliphatic	Permethyl Specialties
	hydrocarbon	
Permethyl 101	Aliphatic	Permethyl Specialties
	hydrocarbon	
Trivent OC-16	Cetyl octanoate	Trivent Chemical
		Company
Cetiol S	Dioctyl	Henkel Corporation
	cyclohexane	
Peceol Isostearique	Glyceryl	Gattefosse
	isostearate	
Monomuls 90-018	Glycerol oleate	Henkel Corporation
Fancol Polyiso 275	Hydrogenated	The Fanning Corp.
	polyisobutene	
Finsolve TN	C12-C15 alcohol	Finetex
	benzoate	
Finsolve SB	Isostearyl	Finetex
	benzoate	
Prisorine 3700	Polyglycerol -3	Unichema North
	Diisostearate	America
Prisorine 3792	Polyglycerol-2	Unichema North
	diisostearate	America
Prisorine 3791	Polyglycerol-2	Unichema North
	monoisostearate	America
Glucate DO	Methyl glucoside	Amercol
·	dioleate	
Glucate SS	Methyl glucoside	Amercol
	sesquistearate	

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Estol 3609	Glycerol tri-2-	Unichema North
ESTOI 3609	Giyceroi cii-2-	
	ethylhexanoate	America
Dow Corning 556	Phenyl	Dow Corning
	tris(trimethylsil	
	oxy) silane	

Trade Name	Chemical Name	Vender		
Ceraphyl 230	Diisopropyl Adipate	ISP Van Dyk Inc		
Mineral oil	Hydrocarbon	Witco		
Novel II 12-5	Ethoxylated alcohol	Condea Vista		
Ethoxylate	or Branched Guerbent ethoxylate	Company		
Cholesterol	Cholesterol	Rita Corporation		
Surfactol Q4	Ricinoleamidopropyl	CasChem		
	dimonium sulfate			
Westchlor 200 50%	Aluminum	West Wood		
w/w	chlorohydrate (ACH)			
Low zirconium penta	Low zirconium:	Reheis		
solution R280-130	Aluminum Zirconium Pentachlorohydrate			
Rezal 67 Solution	Aluminum Zirconium	Reheis		
40%w/w	Pentachlorohydrate (penta)			
Westchlor Zr 44 50%	Aluminum Zirconium	West Wood		
w/w	tetrachlorohydrate (tetra)			
Westchlor Zr 41	Aluminum Zirconium	West Wood		
45%w/w	tetrachlorohydrex- glycine			

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The foregoing description and examples illustrate selected embodiments of the present invention. In light thereof, various modifications would be suggested to one skilled in the art, all of which are within the spirit and scope of this invention.

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Claims:

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- A composition which is selected from the group consisting of a microemulsion, a liquid crystal, or a mixture of a microemulsion and a liquid crystal which comprises an antiperspirant salt, a cosmetic oil, and a combination of at least one cationic quaternary surfactant and at least one nonionic surfactant.
- 10 2. A composition in accordance with claim 1 characterised in that said antiperspirant salt is selected from the group consisting of aluminum, zirconium and mixed aluminum/zirconium salts.
- 15 3. A composition in accordance with claim 1 or 2 characterised in that said antiperspirant salt is a zirconium salt complexed with aluminum salts having coordinated or bound water.
- 20 4. A composition in accordance with any preceding claim characterised in that said antiperspirant salt is present in the aqueous phase at from about 1 to about 60%.
- 25 5. A composition in accordance with claim 4 characterised in that said antiperspirant salt is present in the aqueous phase at from 10% to about 60%.
- A composition in accordance with any preceding claim
 characterised in that said aqueous phase further
 comprises a buffer, a qlycol, a sugar, a cyclodextrin,

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a preservative, an antimicrobial, a chelating agent, a water-soluble polymer, an anticholinergic, a monovalent salt, a divalent salt, a trivalent salt, fragrances or mixtures thereof.

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 A composition in accordance with any preceding claim cit said aqueous phase is present at about 1% to about 60%, more preferably at 5% to 30%, and most preferably at 10 to 25%.

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- 8. A composition in accordance with any preceding claim characterised in that said cosmetic oil comprises esters, ethers, long chain alcohols or ethoxylated alcohols, hydrocarbons, fatty acids, monoglycerides, diglycerides triglycerides, fragrances and volatile or non-volatile silicone fluids, and cholesterol.
- A composition in accordance with claim 8 characterised in that said oil phase comprises silicone fluids which in turn comprise a volatile or non-volatile silicone such as cyclomethicone or dimethicone.
 - 10. A composition in accordance with claim 8 or 9 characterised in that said non-volatile silicone is phenyl tris(trimethylsiloxy) silane.
 - 11. A composition in accordance with claim 8 characterised in that said esters are selected from the group consisting of cetyl octanoate, C12 -15 alcohol benzoate, isostearyl benzoate, diisopropyl adipate and mixtures thereof.

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- 12. A composition in accordance with claim 8 wherein said hydrocarbon fluids are selected from the group such as aliphatic hydrocarbons; hydrogenated polydecenes; hydrogenated polybutenes; dioctylcyclohexane; mineral oil, cyclohexane and mixtures thereof.
- 13. A composition in accordance with any preceding claim characterised in that the cationic quaternary ammonium surfactant has the following structure:

R-CO-NH-
$$(CH_2)_n$$
-N⁺- $(CH_2)_z$ -CH₃ A⁻

wherein n is one to six.

- 15 x is zero to three
 - v is zero to three
 - z is zero to three
 - with the proviso that $x+y+z \le 6$
 - A is any physiologically acceptable counter ion which does not adversely affect the composition, and more specifically A can be selected from the group consisting of chloride, bromide, ethosulfate, methyl sulfate, lactate, acetate, nitrate or sulfate.
 - where R is a ricinoleic derivative:
- 25 CH₃ (CH₂) ₅CH(OH) CH₂-CH=CH- (CH₂) ₇-Or mixtures thereof.
 - 14. A composition in accordance with claim 13 wherein n=3, x=1, y=0, z=0, A = ethosulfate and R = CH₃-(CH₂)₅. CH(OH)-CH₂-CH=CH-(CH₂)₇-.

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- 15. A composition in accordance with any preceding claim characterised in that said cationic quaternary ammonium surfactant is present at 0.1% to 30%, more preferably at 1% to 30%, most preferably at 2% to 15%.
- 16. A method for controlling or preventing underarm perspiration and malodor which comprises applying, to an underarm, an effective amount of a composition of claim 1.

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1/24766 A

(54) Title: ANTIPERSPIRANT COMPOSITIONS COMPRISING MICROEMULSIONS

(57) Abstract: Stable, clear, antiperspirant microemulsions containing cosmetic oils, antiperspirant salts, and water and combinations of cationic quaternary ammonium salt are provided. These microemulsions can be used in different types of applicators such as roll-on, sponge, mouses, pad, brush, gel and aerosol or non-aerosol spray applicators.



. 12	ŭ .							
COMBINED SCLARATION FOR International Applications	ATENT APPLICATION	ATENT APPLICATION AND POWER OF ATTORNEY (Includes Reference to PCT Attorney Docket No. J6547(C)						
As a below named inventor, I hereby declare that:								
My residence, post office address and citizenship are as stated below next to my name.								
I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patient is sought on the invention entitled:								
ANTIPERSPIRANT COMPOSITIONS COMPRISING MICROEMULSIONS								
the specification of which (check only one item below):								
is attached hereto.								
☐ was filed as United States appli	cation Serial No. 09/_	or	n and	was amended on	(if app	licable)		
was filed as PCT international	application PCT/EP00	09144 on Se	ptember 18, 2000	and was amended	under PCT Article 19 on	(if ap	plicable)	
I hereby state that I have reviewed	and understand the co	ntents of the	above-identified sp	ecification, includi	ng the claims, as amende	ed by any am	endment ref	erred to above.
I acknowledge the duty to disclose	information which is m	itenal to the p	patentability of this	application in acco	ordance with Title 37, Coo	ie of Federal	Regulations	, § 1.56(a).
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PRIOR FOREIGN/PCT APPLICAT	TION(S) AND ANY PR	ORITY CLAIR	MS UNDER 35 U.S	.C. 119:				
COUNTRY (if PCT, indicate "PCT") APPLICAT	APPLICATION NUMBER			DATE OF FILING (day, month, year)		PRIORITY CLAIMED UNDER 35 U.S.C. 119	
USA	60/157,382	60/157,382			1 OCTOBER 1999		YES	
hereby claim the benefit under Title 35, United States Code §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are isled below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title St. Sultied States Code §112. Lackowskide; the duty to disclose material information as defined in Title 37, Code of Federal Regulations §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application. PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. 120.								
U.S. APPLICATIONS				STATUS (CHECK ONE)				
U.S. APPLICATION NUMBER	ER U.S. FILING DATE		NG DATE		PATENTED	PENDING		ABANDONED
PCT APPLICATIONS DESIGNATING THE U.S.								
PCT APPLICATION NO.	PCT FILING DATE	ILING DATE U.S SERIAL NI ASSIGNED (if a						
PCT/EP00/09144	18 SEPTEMBER 20	PTEMBER 2000						
PRIOR U.S. PROVISIONAL APPL	ICATION(S) FOR BEI	EFIT UNDER	R 35 U.S.C. 119(e)					
APPLICATION NUMBER			DAT	DATE OF FILING (day, month, year)				

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Attorney Docket No.

COUNTRY OF CITIZENSHIP

STATE & ZIP CODE/COUNTRY

J6547(C)

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agents(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (List name and registration in the Patent and Trademark Office connected therewith. CUSTOMER NUMBER: 000201 Direct all correspondence to : CUSTOMER NUMBER 000201 C BADEMARY FULL NAME OF INVENTOR FAMILY NAME FIRST GIVEN NAME SECOND GIVEN NAME MA ZHUNING RESIDENCE AND CITY STATE OR FOREIGN COUNTRY COUNTRY OF CITIZENSHIP CITIZENSHIP CHICAGO ILLINOIS USA POST OFFICE POST OFFICE ADDRESS CITY STATE & ZIP CODE/COUNTRY. ADDRESS C/O UNILEVER HOME & PERSONAL CHICAGO ILLINOIS 60008, USA CARE USA: 3100 EAST GOLF ROAD. ROLLING MEADOWS FULL NAME OF INVENTOR FAMILY NAME FIRST GIVEN NAME SECOND GIVEN NAME BRUCKS RICHARD MADK RESIDENCE AND STATE OR FOREIGN COUNTRY COUNTRY OF CITIZENSHIP CITIZENSHIP CHICAGO ILLINOIS USA POST OFFICE ADDRESS POST OFFICE CITY STATE & ZIP CODE/COUNTRY ADDRESS C/O UNILEVER HOME & PERSONAL CHICAGO ILLINOIS 60008, USA CARE USA, 3100 EAST GOLF ROAD, ROLLING MEADOWS 203 FULL NAME OF FAMILY NAME FIRST GIVEN NAME SECOND GIVEN NAME INVENTOR

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (Includes Reference to PCT

International Applications)

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that sixth willful false statements may pioporatize the validity of the application or any patent sixung hieron.

CITY

STATE OR FOREIGN COUNTRY

SIGNATURE OF INVENTOR 201	SIGNATURE OF INVENTOR 202 Ruberd Mark Bruches	SIGNATURE OF INVENTOR 203
DATE 5/15/02	DATE May 15, 2002	DATE